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Characterization of Sidewall and Planar Surfaces of Electroformed LIGA Parts

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Abstract

The nature of surfaces and the way they interact with each other during sliding contact can have a direct bearing on the performance of a microelectromechanical (MEMS) device. Therefore, a study was undertaken to characterize the surfaces of LIGA fabricated Ni and Cu components. Sidewall and planar surfaces were examined by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Surface roughness was quantified using the AFM. Post-processing (e.g. lapping, removal of polymer film) can profoundly influence the morphology of LIGA components. Edge rounding and smearing of ductile materials during lapping can result in undesirable sidewall morphologies. By judicious selection of AFM scan sizes, the native roughness (~10 nm RMS) can be distinguished from that arising due to post processing, e.g. scratches, debris, polymer films. While certain processing effects on morphology such as those due to lapping or release etch can be controlled, the true side wall morphology appears to be governed by the morphology of the polymer mold or by the electroforming process itself, and may be much less amenable to modification.

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Introduction

This report describes morphological studies to date on LIGA components fabricated at Sandia National Laboratories in New Mexico (pure Ni) and California (pure Ni and pure Cu). Sidewall and planar surfaces were characterized using scanning electron microscopy (SEM) and atomic force microscopy (AFM). Energy dispersive x-ray spectra were obtained to examine the purity of metal parts. The majority of the studies were performed on LIGA gear teeth. It must be noted that no batch or serial numbers were assigned to the components so that correlating the surface morphology of a particular component to its processing history was not feasible for this analysis.

Experimental Details

As shown schematically in Fig.1, LIGA parts were mounted to the side of a square metal block with conducting carbon tape so that the same parts could be examined by SEM as well as the AFM. This procedure also enabled the sidewalls to be accessed by the AFM tip for imaging. By rotating the specimen block 90 degrees, planar surfaces could be imaged. LIGA parts, in as-received condition, were analyzed in the Hitachi S-4500 field emission SEM equipped with an energy dispersive x-ray spectrometer. SEM imaging was done at 5 kV operating voltage while EDS analysis was performed at 15 kV. Atomic force microscopy was performed using a Park Scientific Instruments Autoprobe LS. A ThermoMicroscopes silicon ultralever was used in contact mode in an AFM/LFM microscope head. All parts were imaged in ambient air and the as-received condition. Tip shape was not deconvoluted from the images, and likely caused some modification of images taken with scans of 1 μ m or less in size. This distortion appears as round shapes

superimposed on any feature that has dimensions on the order of the tip size (10nm radius of curvature) or smaller.

Results and Discussion

Morphology of Lapped Surfaces

Metallographic polishing of softer metals is known to be a challenging task as there will be some stress concentration at the unsupported edges of a lapped component, e.g. gear teeth. This is clearly illustrated in the SEM micrographs of a LIGA Cu gear shown in Fig. 2. Damage to gear teeth edges is evident in Fig. 2a. Scratches are present on the lapped surface (Fig. 2a). Smearing of material on teeth edges, presumably from lapping, can be seen in Fig. 2b. Figure 2c is a higher magnification micrograph showing extensive plastic deformation of copper during lapping that actually flowed beyond the sidewall, and was probably bent down after release. Scratches seen on gear teeth at the location marked "X" in Fig. 2a were probably due to uncontrolled handling of parts prior to analysis. Edge damage to gear teeth does not appear to be a major problem in the case of LIGA Ni gears. At moderate magnifications, the lapped surface appeared to be smooth and free of scratches, as can be seen from Fig. 3b. Lapping on this particular Ni gear introduced only submicron scratches (Fig. 3b and 3c).

Morphology of Bottom Surfaces

Typical SEM micrographs of a LIGA Ni gear on the release side are shown in Fig. 4. At higher magnifications, the surface revealed a honeycomb type of granular structure with grain size in the nanometer range (Fig. 4b). SEM micrographs of the release surface of a

Cu gear are given in Fig. 5. It appears that a residual layer of the polymeric film was still intact on this particular surface.

Sidewall Morphology

Figure 6 is a set of SEM images of a Cu part sidewall showing incomplete removal of the polymeric film. However, the patch in Fig. 6c revealed the true sidewall morphology. The parts were subjected to a release process to dissolve the PMMA material after electroforming and lapping [1]. Issues such as incomplete removal of the mold material from narrow crevices, or redeposition after dissolution, should be addressed with researchers in process development to ensure that surfaces are clean enough to accept a coating.

An SEM micrograph of a LIGA Ni gear sidewall is shown in Fig. 7. The damage seen at locations marked "X" in Fig. 7 was probably caused by handling of parts prior to analysis. The top is the lapped side showing minor damage to the tooth edge. Higher magnification images of the regions marked "A" and "B" are shown in Fig. 8. There is a blister-like morphology at the top of the gear tooth. The middle is more like a columnar structure (Fig. 8b). The microstructure of electroformed Ni is typically columnar in nature. For instance, earlier work by Buchheit and coworkers [2,3] in this laboratory showed that LIGA Ni has a (100) texture, i.e., (100) crystal orientations are nearly parallel to the growth direction. However, the columnar grain size determined by backscattered electron Kikuchi patterns (BEKP) in those studies [2,3] is much larger than the nanometer-size columns seen in Fig. 8b. It is very likely that the morphology seen in Fig. 8b is a surface feature resulting from the x-ray mask. Examination of mold surfaces

prior to plating could resolve whether the sidewall roughness is due to mold structure. SEM micrographs of another LIGA Ni gear sidewall showing essentially the same features are given in Fig. 9. The LIGA Cu gear sidewalls also showed similar morphology (Fig. 10). The root of the LIGA Ni gear showed steps at regular intervals (Fig. 11a). Similar images from another Ni part (Fig. 11 b and c) show "scales" that appear to be partially detached from the remainder of the gear tooth at one edge. Additional metallographic sectioning and analysis will be required on future samples to determine if this is the case.

SEM examination revealed that LIGA fabricated parts could have various sidewall morphologies. While certain morphological features (e.g. edge rounding and scratches) could easily be controlled by processing, the native sidewall morphology (i.e. columnar texture) may be less amenable to modification. The exact ramifications of columnar texture or fish scales on the tribological behavior are difficult to predict at this stage. However, mechanical interlocking of asperities such as those shown in Figs. 10 and 11 could potentially increase the friction between sliding surfaces. Such features may also influence the local stress distribution and the wear particle generation, which in turn would effect the tribological behavior.

Energy Dispersive Spectroscopy Analysis

Typical energy dispersive x-ray spectra collected from Ni and Cu LIGA parts are given in Figs. 12 and 13 respectively. The spectra essentially showed no impurities. However, it is not possible to determine whether lighter elements such carbon coming from the

residual polymer film were present because the Be window detector is not capable of light element analysis. Scanning Auger analysis will be performed on future samples.

Atomic Force Microscopy of Sidewalls

The sidewall, lapped, and release surfaces of the LIGA parts analyzed above were further probed with the Atomic Force Microscope. AFM was used to quantify the surface roughness of interesting features identified in the SEM. Table 1 compares the RMS roughness of the sidewall, lapped, and release surfaces of three LIGA parts; a copper gear, a Ni square, and a Ni Sandia Logo. Each part was examined at two scan sizes. The larger scan size was chosen to include features such as scratches, while the smaller scan size was chosen to represent the native surface morphology. Because of the shape of some sidewall features the scan sizes for these areas may be different. Note that on some surfaces, the two roughness numbers differ considerably because the larger scan includes scratches, while the smaller scan does not.

Figure 14 shows the tip of a copper gear tooth. For the three parts listed, this sidewall surface was the smoothest. SEM analysis showed that the copper gear is covered with what appears to be a polymer film. The roughness measured on this part is likely characteristic of this film and not the LIGA copper surface. The sidewall surfaces of both Ni parts were similar and exhibited comparable roughness numbers. Figure 15 shows the sidewall of a Ni Sandia Logo (NM). Roughness is present on this surface at two scales. Undulations with a period of $\sim 5\mu\text{m}$ perpendicular to the thickness direction can be seen in the $20\mu\text{m}$ scan. This type of feature was noticed on all the Ni LIGA parts, and may be due to a roughness in the patterned PMMA resist. A much smaller (nm scale) roughness

can be seen in the 5 μ m scan. This roughness is the columnar structure seen in the SEM (Figure 10). Because these features are on the order of the tip radius they are convoluted with the tip shape in the image.

Figure 16 shows typical lapped and release surfaces of Ni LIGA parts. Although considerable scratches can be seen on the lapped surfaces, their RMS roughness suggests that the scratches are not very deep. The release surfaces appear to be more uniform than the lapped surfaces, however, the roughness data suggests that the features on this surface are quite deep. The roughness of the lapped and release sides of the copper parts is large (order 1-10 μ m). Again, this is thought to be characteristic of the polymer film covering the surface of these parts, and not the copper itself. Interestingly, roughness data for this film suggest that it is smooth on the sidewall and rough on the lapped and release surfaces. The large roughness seen on the lapped and release surfaces is characteristic of holes in the film, debris on the film surface, and not the film itself (see SEM image Fig. 5b).

We have demonstrated that the combination of AFM and SEM is an effective way to characterize LIGA surfaces. Large areas can be rapidly surveyed with the SEM, allowing features of interest to be identified for later examination with the AFM. SEM also permits elemental analysis of the surface. AFM allows surface roughness to be quantified. Careful selection of scan size allows native roughness to be distinguished from that due to post processing, e.g. scratches, debris, polymer films, etc. The detailed surface knowledge provided by AFM will be critical to development of surface coatings which have the desired physical interlocking with the LIGA surfaces.

Summary and Conclusions

The current study has highlighted the importance of surface characterization before conducting tribological performance evaluations with LIGA parts, such as in the Tribology Test Vehicle or the LIGA Friction and Wear Tester. The surfaces of parts, notably the gear teeth, must be characterized prior to assembling the vehicle. Issues concerning edge damage to gear teeth and incomplete removal of polymer films could be solved by careful control of the post processing steps, namely lapping/metallographic polishing and release etch. Atomic Force Microscopy is a powerful tool in quantifying the sidewall roughness of LIGA MEMS components. A specimen tracking system should be implemented so that meaningful correlation between processing and surface morphology may be made.

Acknowledgements

Valuable discussions with Todd Christenson (01743), Steve Leith (formerly with 08729) and Carl Vanecek (02613) are gratefully acknowledged. The authors wish to thank Wendy Cieslak for reviewing this report.

References

1. T. A. Christenson, in: Recent Advances in MEMS/MOEMS Technologies, Chapter V: Microsystems Manufacturing Technologies, SPIE Handbook, to be published.
2. T. E. Buchheit, D. A. Lavan, T. R. Christenson, S. Leith and J. Michael, "Microstructural and Mechanical Properties Investigations of Electrodeposited and Annealed LIGA Ni Structures," in preparation.
3. T. R. Christenson, T. E. Buchheit, D. T. Schmale and R. T. Boureier, "Mechanical and Metallurgical Characterization of LIGA Fabricated Ni and 80Ni-20Fe Permalloy" In: *Microelectromechanical Structures for Materials Research*, Proceeding MRS Spring Meeting, MRS, Warrendale, PA, 1998.

Table 1
Summary of AFM analyses on LIGA parts

Sidewall

Part Description	Scan Size (μm)	RMS Roughness (nm)
Cu Gear tooth tip (CA) (Fig. 14)	40	18.8
Cu Gear tooth tip (CA)	1	1.6
Ni Square (CA)	50	27
Ni Square (CA)	10	9.2
Ni Sandia Logo (NM) (Fig. 15a)	20	21
Ni Sandia Logo (NM) (Fig. 15b)	5	11

Lapped Surface

Part Description	Scan Size (μm)	RMS Roughness (nm)
Cu Gear (CA)	40	198
Cu Gear (CA)	10	120
Ni Square (CA)	40	15
Ni Square (CA)	10	8.0
Ni Sandia Logo (NM) (Fig. 16a)	40	17
Ni Sandia Logo (NM)	10	14

Release Surface

Part Description	Scan Size (μm)	RMS Roughness (nm)
Cu Gear (CA)	40	155
Cu Gear (CA)	10	97
Ni Square (CA)	40	29
Ni Square (CA) (Fig. 16b)	10	4.9
Ni Sandia Logo (NM)	40	135
Ni Sandia Logo (NM)	10	51

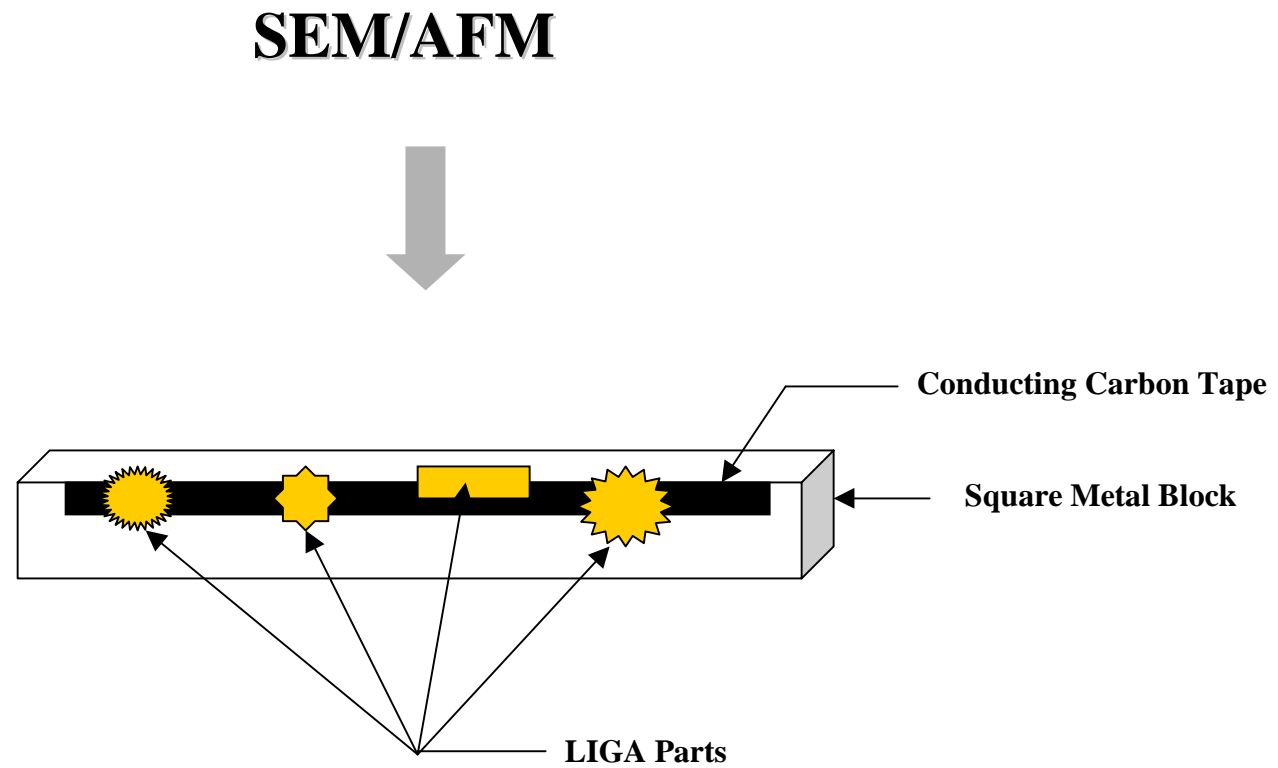


Fig. 1 Schematic illustration of SEM/AFM specimen (LIGA parts) mounting for examining the sidewall morphology.

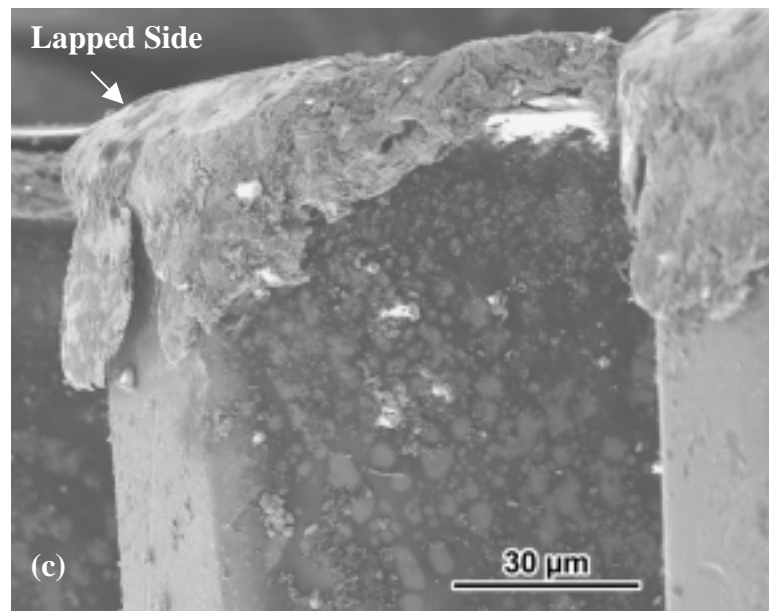
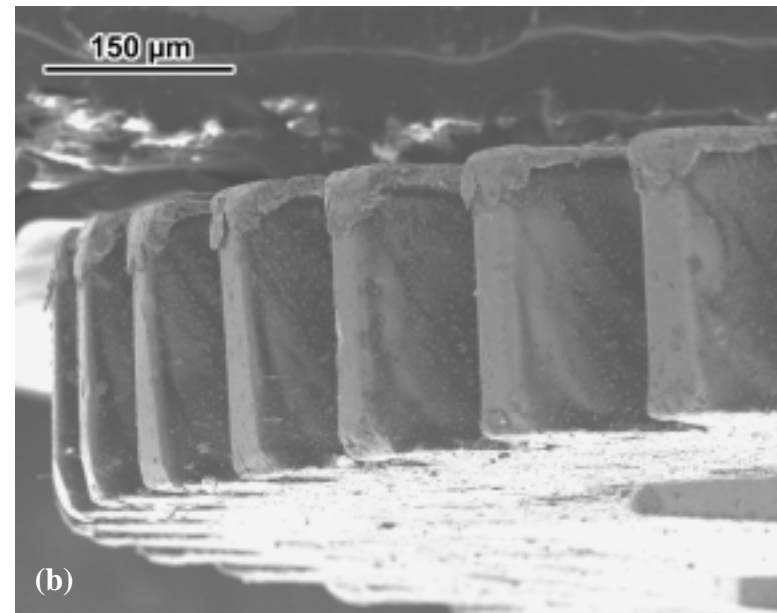
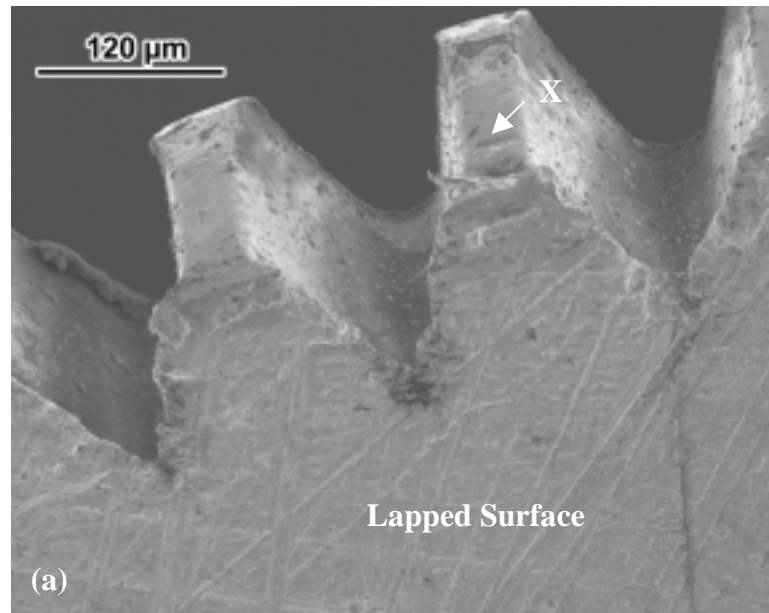


Fig. 2 SEM images of a LIGA Cu (CA) gear showing edge rounding (or lack of edge retention).

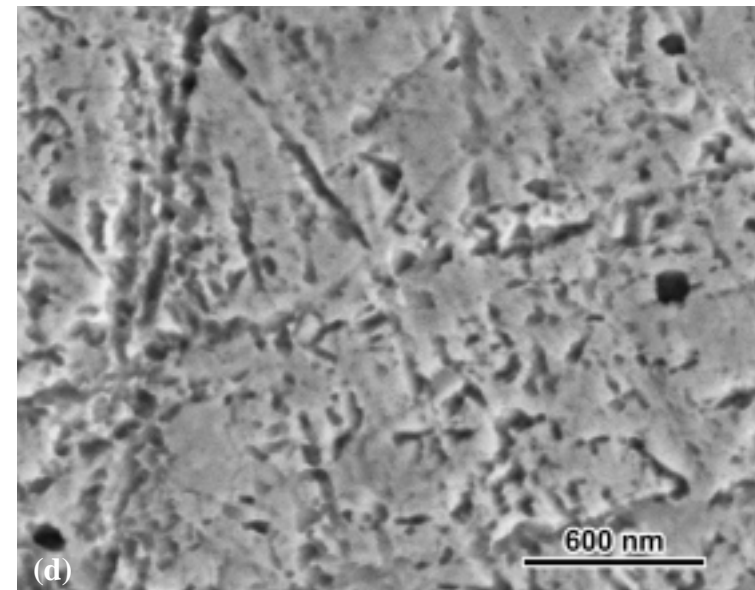
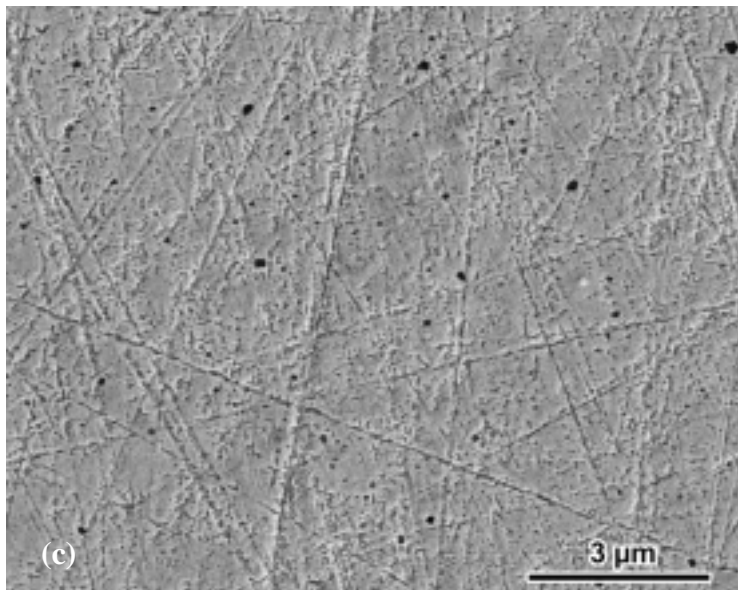
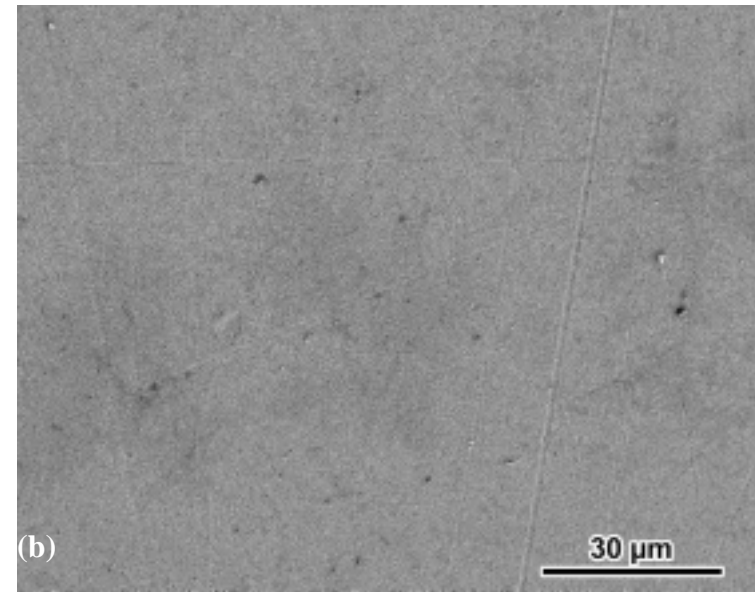
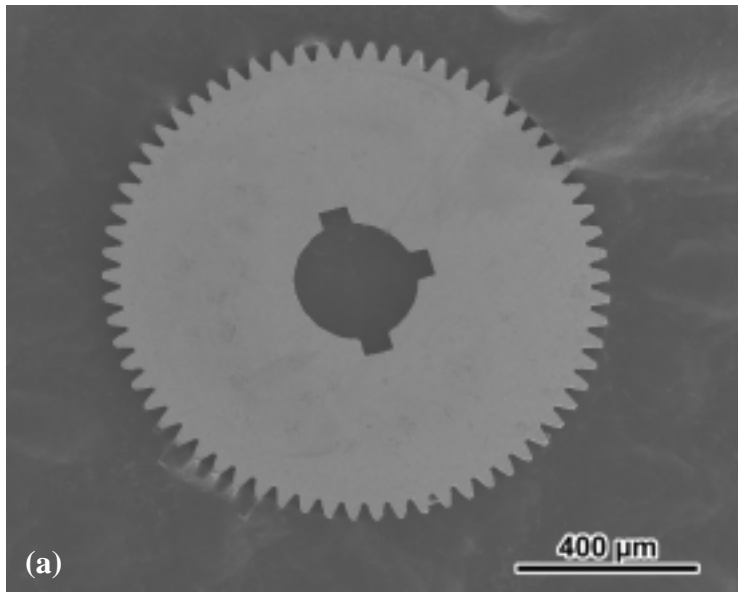


Fig. 3 Typical surface morphology of a LIGA Ni gear (NM): Lapped surface.

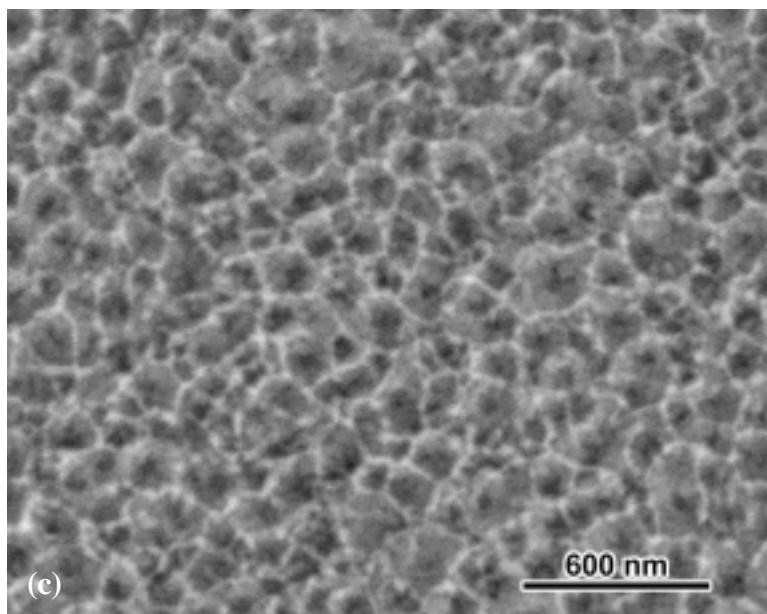
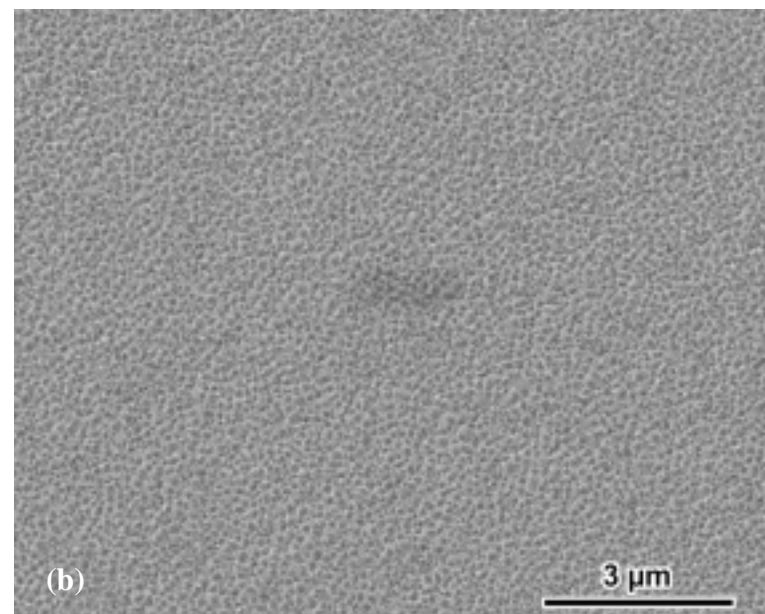
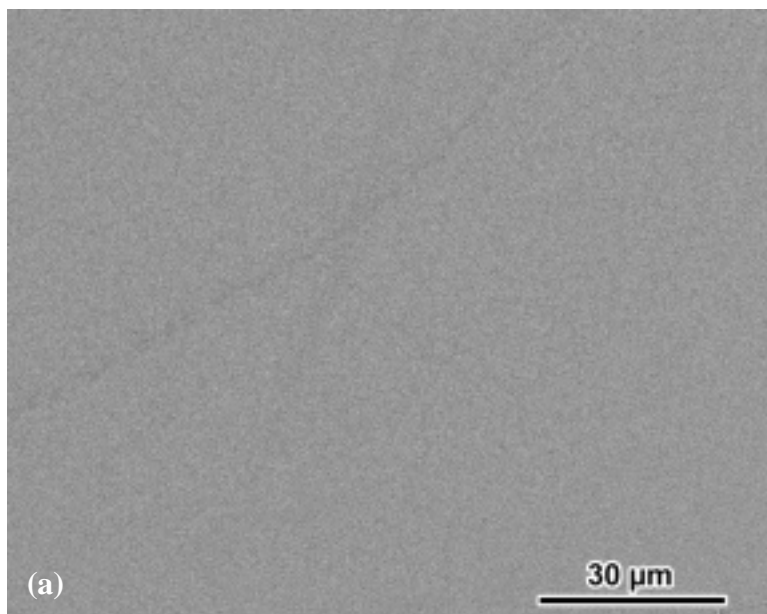


Fig. 4 Surface morphology on the release side: LIGA Ni gear (NM).

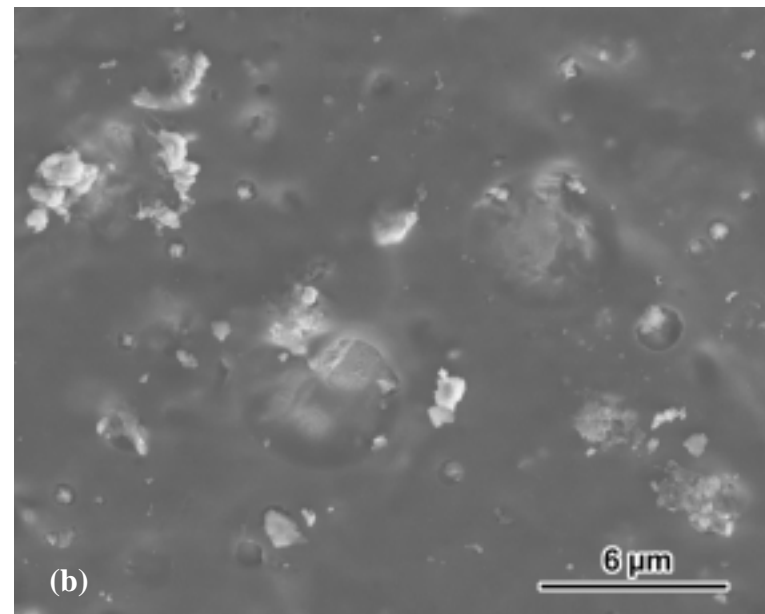
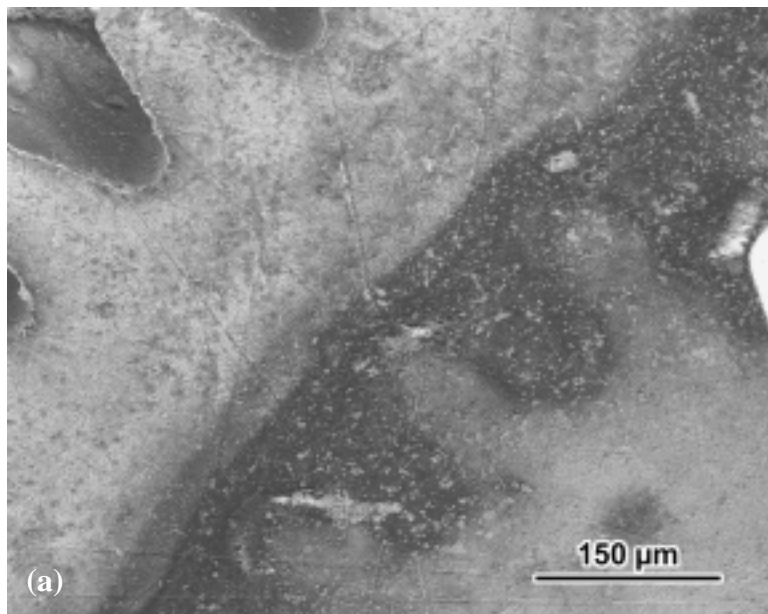


Fig. 5 Surface morphology on the release side of a Cu gear (CA).

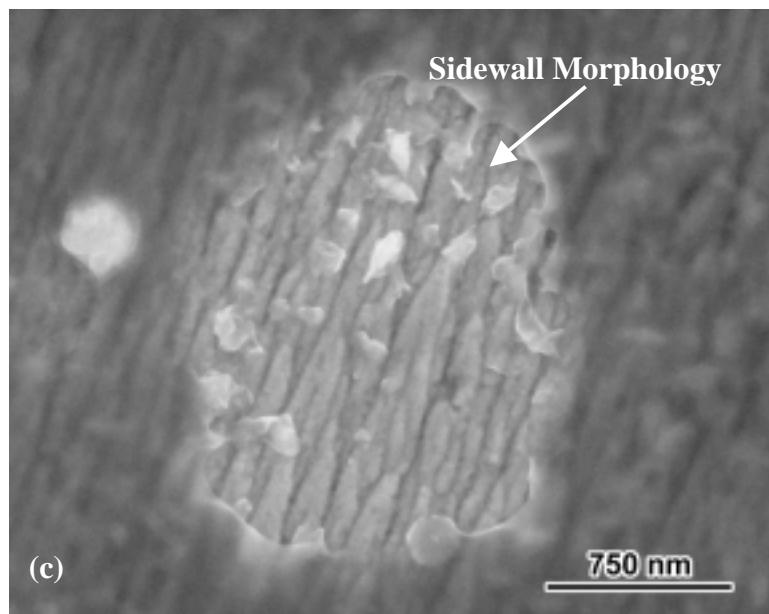
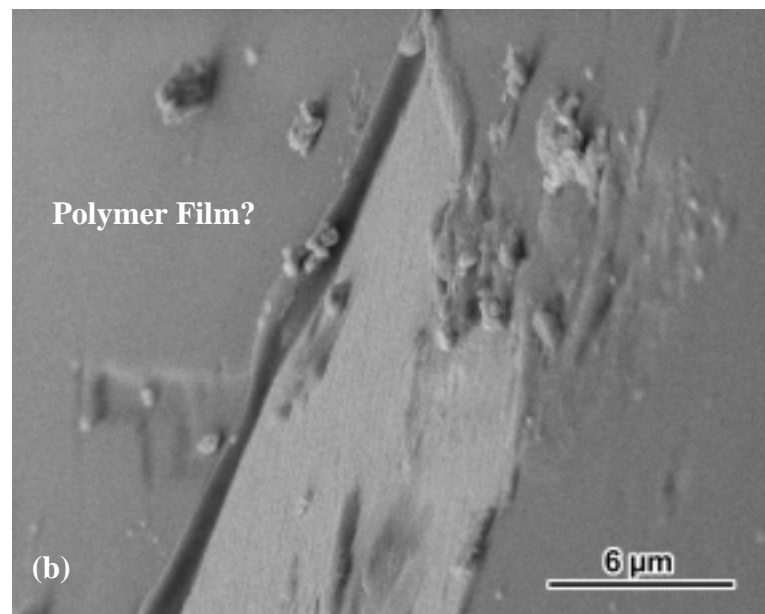
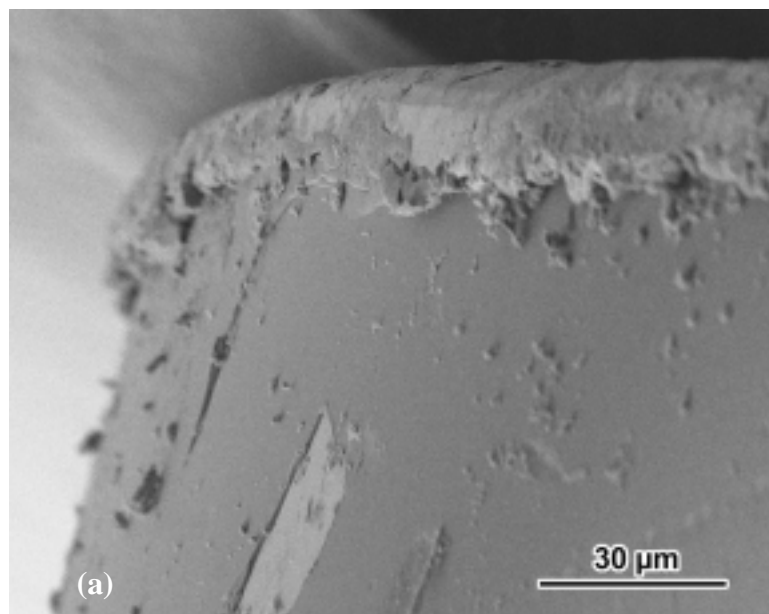


Fig. 6 SEM images showing incomplete removal of polymer film on the sidewall of a LIGA Cu gear (CA). True sidewall morphology can be seen in the patch in Fig. (c).

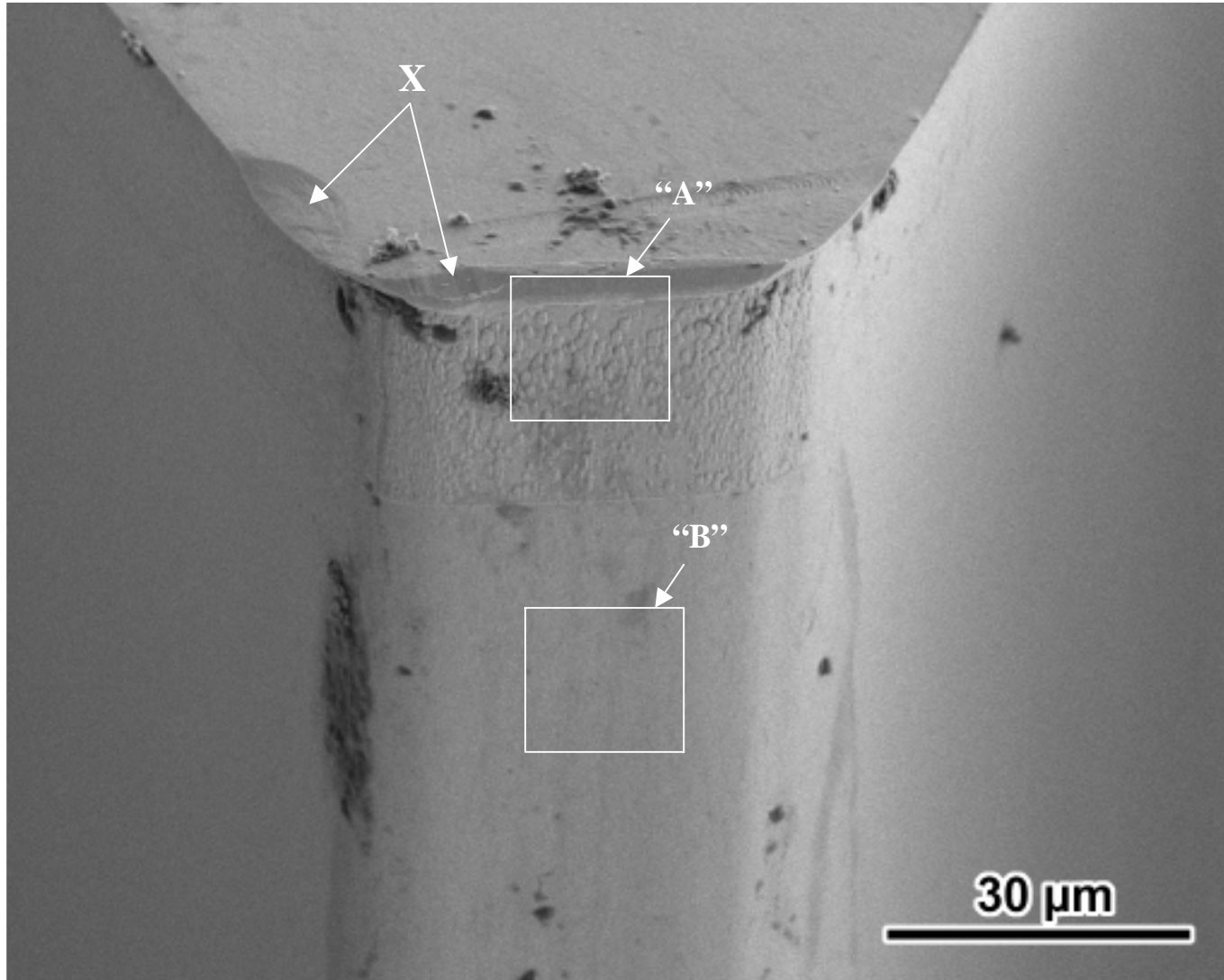


Fig. 7 Sidewall morphology of a Ni Gear (NM). Higher magnification images of regions marked "A" and "B" are given in Fig.7.

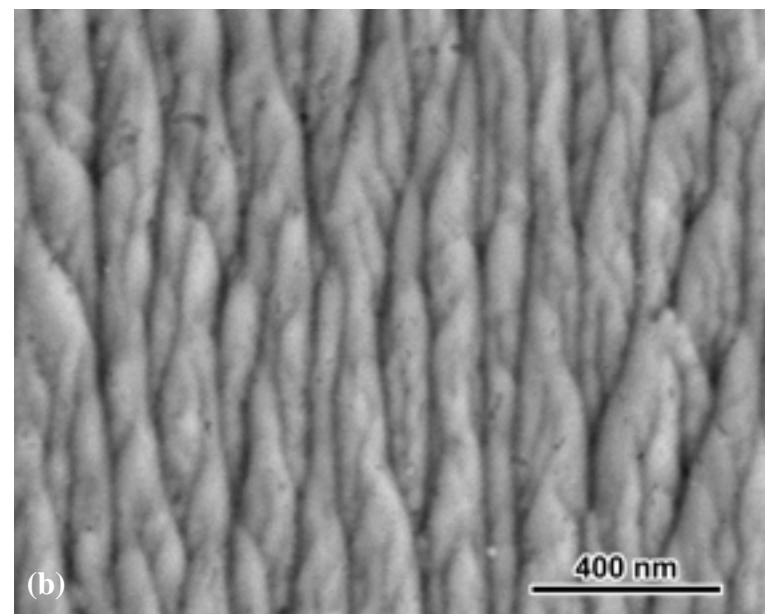
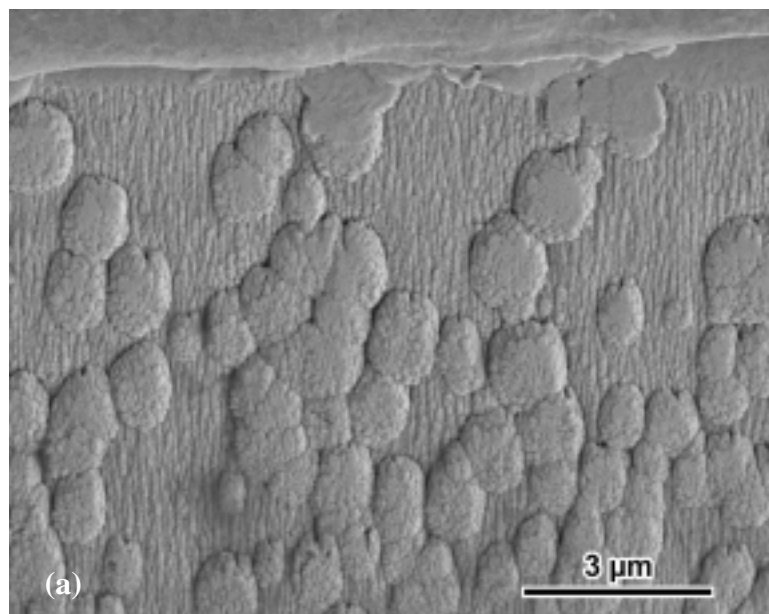


Fig. 8 Higher magnification images of sidewall morphology of a LIGA Ni gear (NM). Figure (a) corresponds to the region marked “A” in the previous figure, while Fig. (b) corresponds to the higher magnification image of the region marked “B” in Fig. 7.

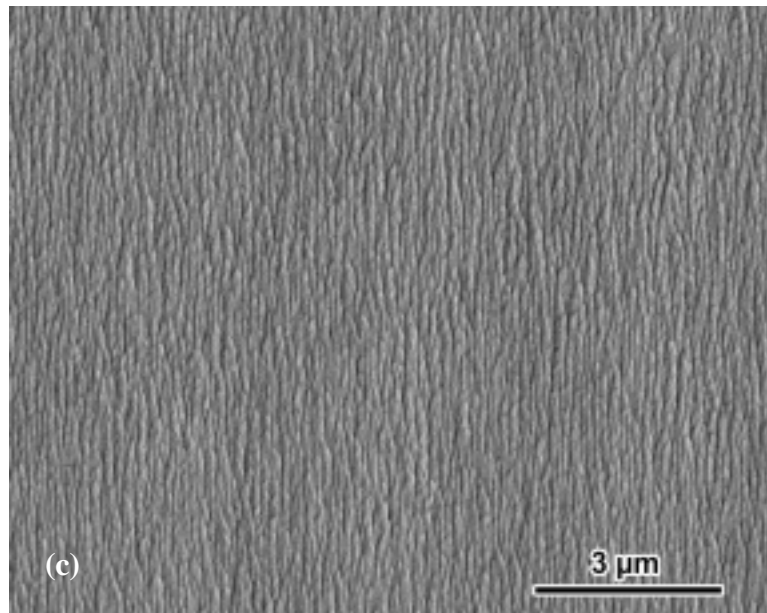
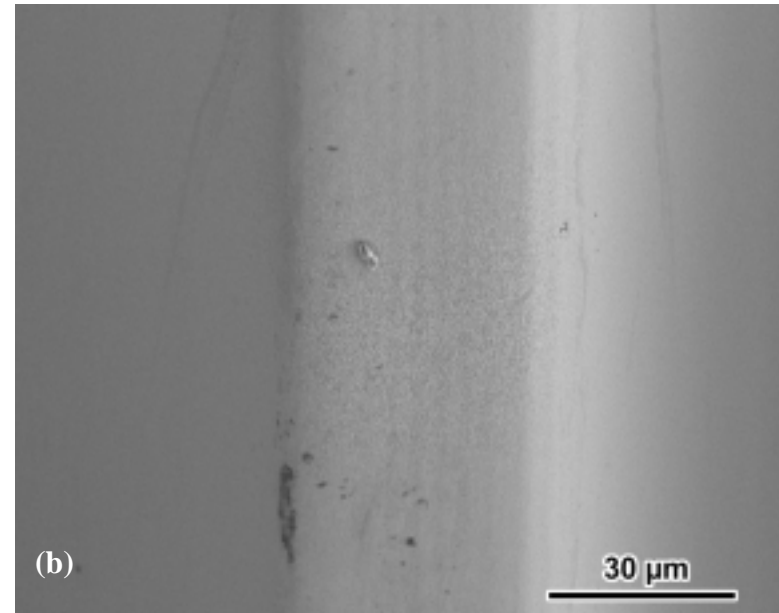
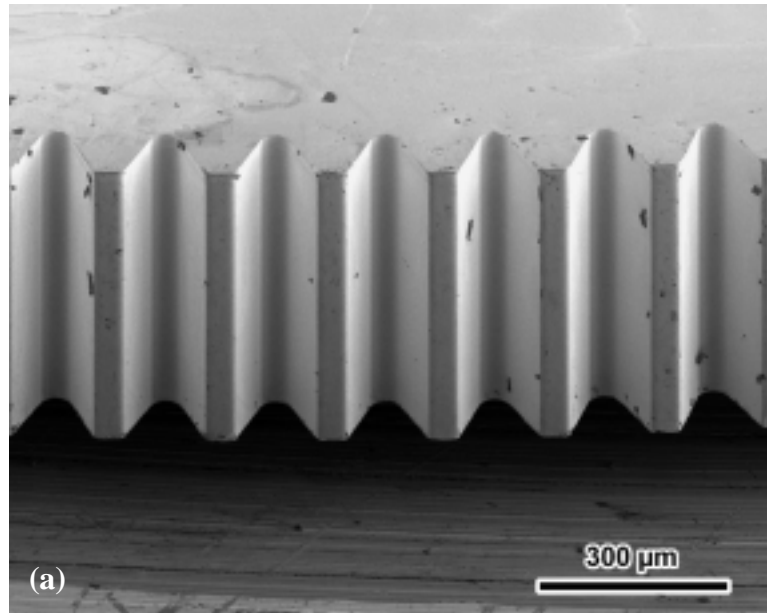


Fig. 9 Sidewall morphology of another Ni Gear (NM).

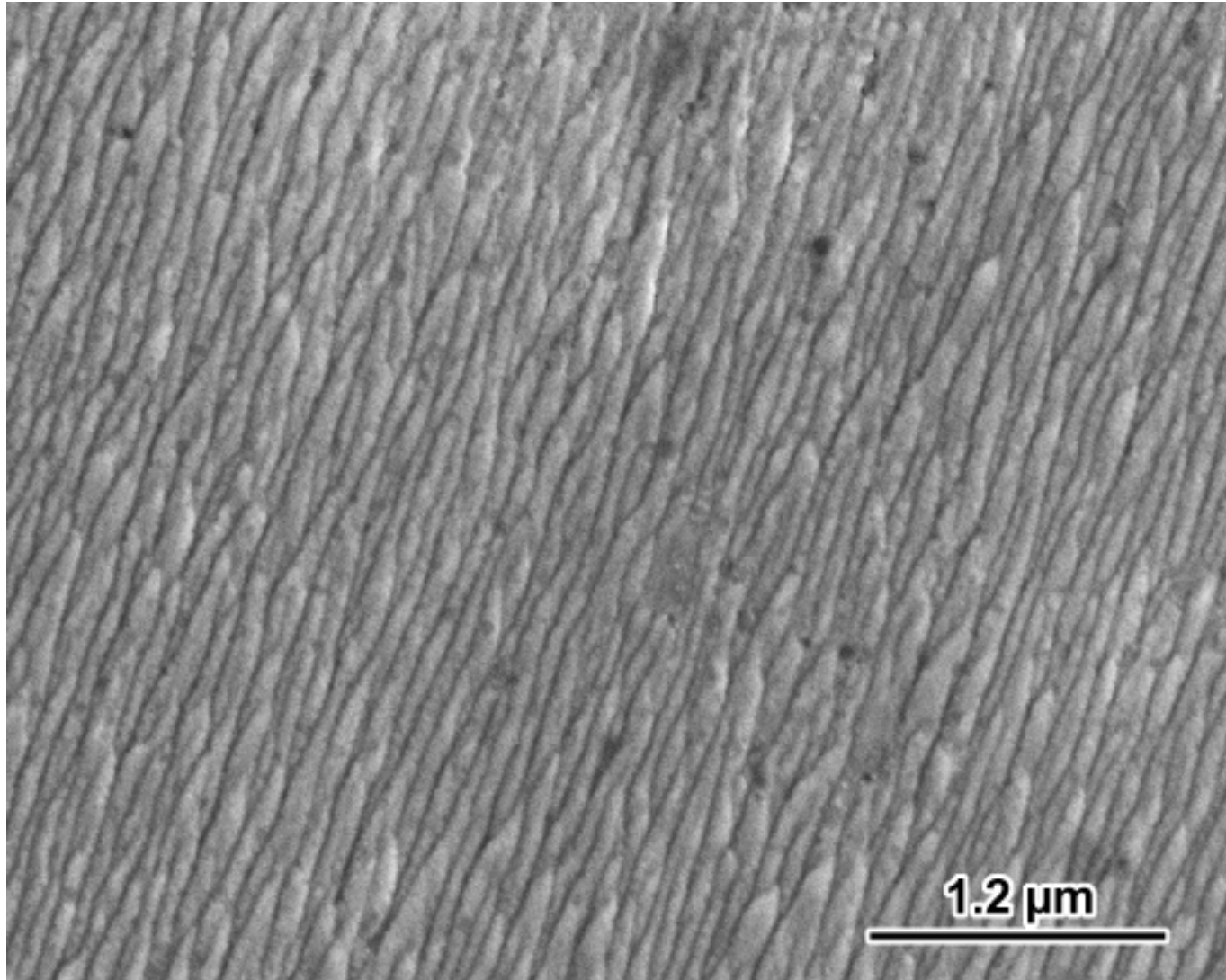


Fig. 10 Side wall morphology a LIGA Cu gear (CA).

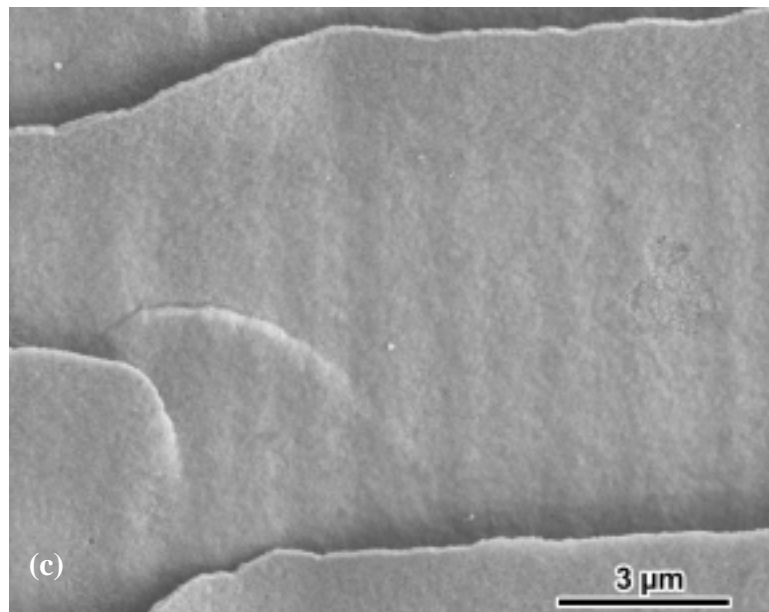
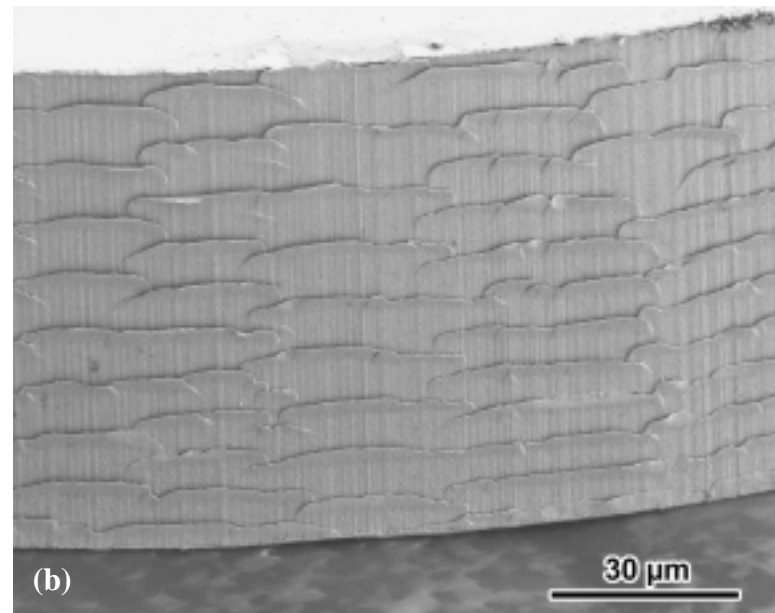
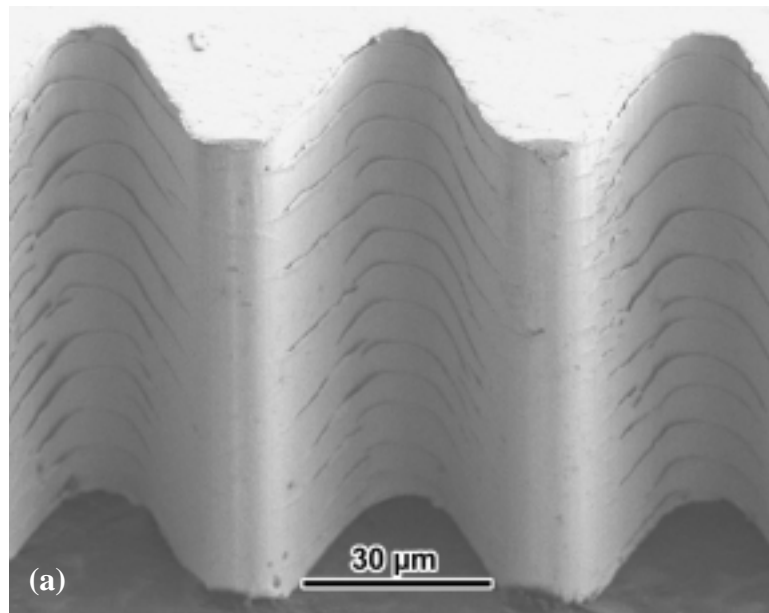


Fig. 11 (a) and (b) Sidewall morphologies of two different LIGA Ni (NM) gears; (c) higher magnification micrograph of Fig. (b).

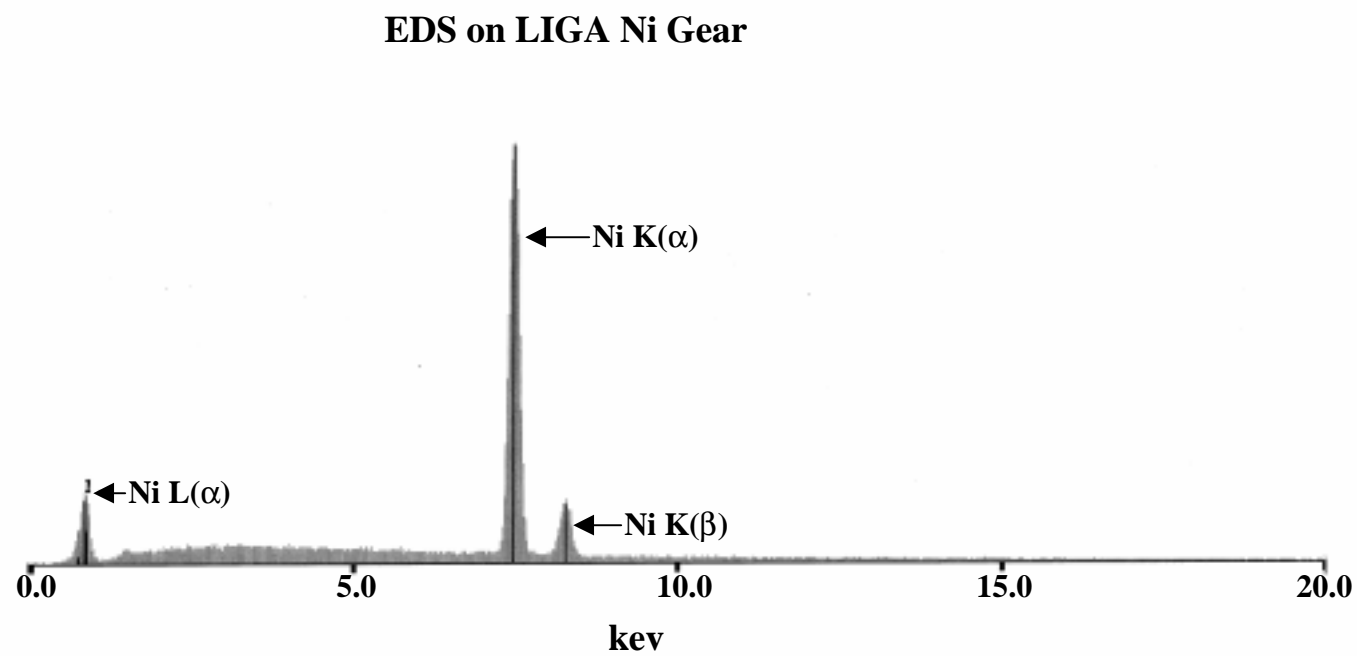


Fig. 12 A typical energy dispersive x-ray spectrum (EDS) collected from a LIGA Ni (NM) gear.

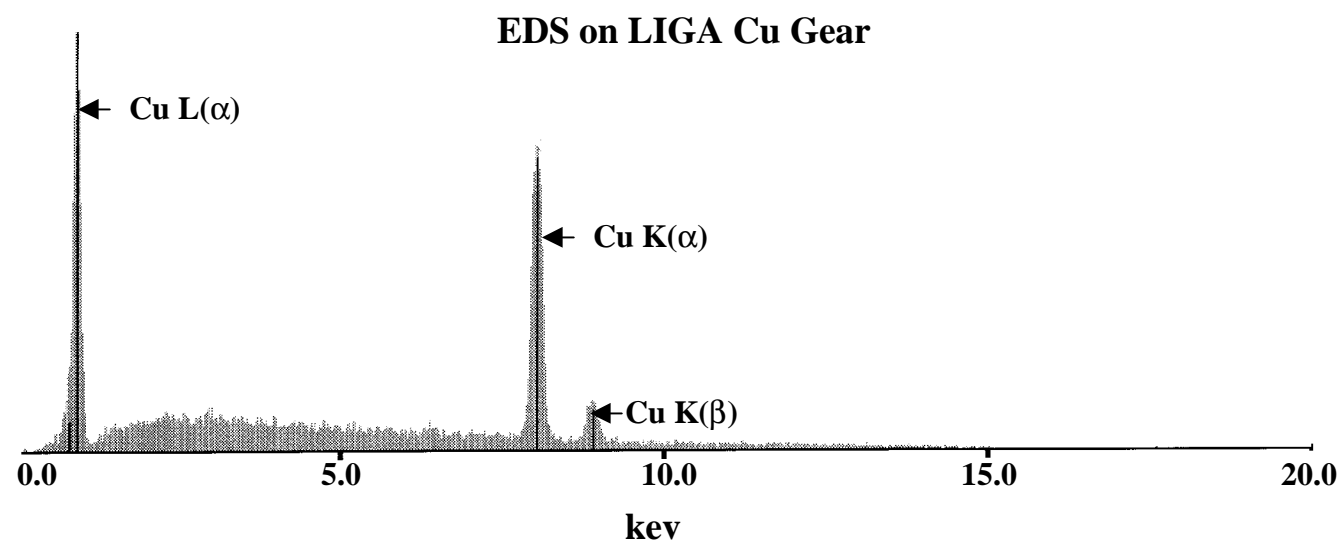


Fig. 13 A typical energy dispersive x-ray spectrum (EDS) collected from a LIGA Cu (CA) gear.

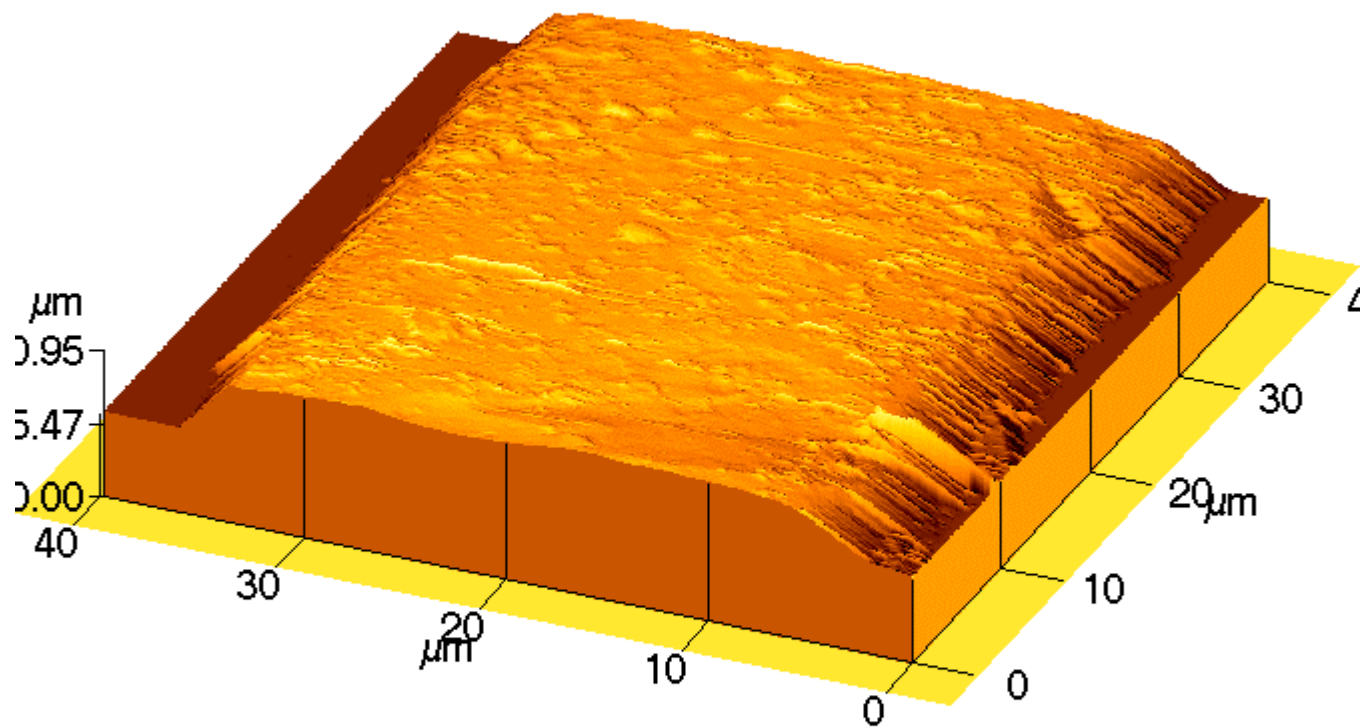


Fig. 14 Tip of a Cu gear (CA) tooth: AFM image.
A location such as this can be seen in Fig. 2b.

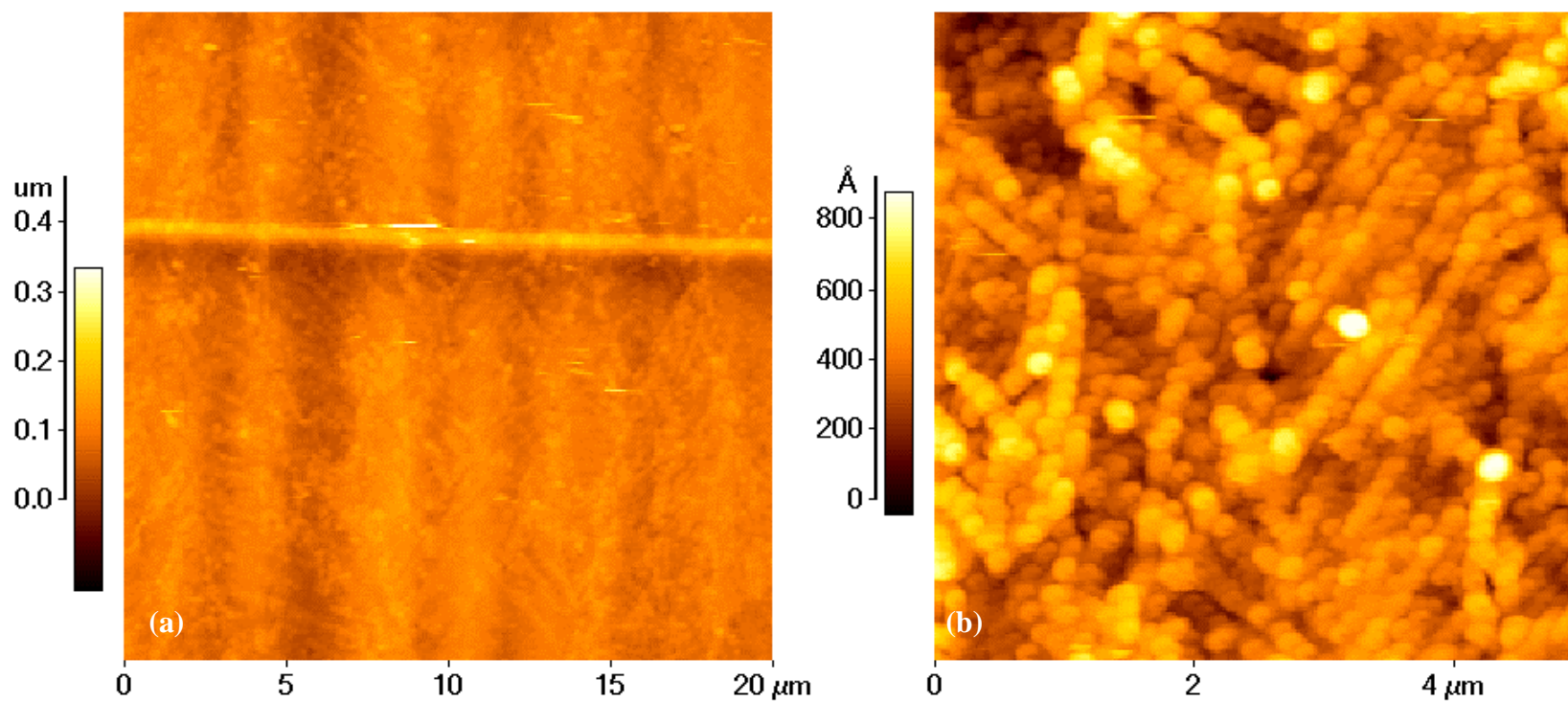


Fig. 15 Sidewall morphology of a LIGA Ni (NM) SANDIA LOGO: AFM images. (a) showing surface undulations, (b) showing the columnar structure seen in Fig. 10. Distortion in the image is an effect of tip convolution.

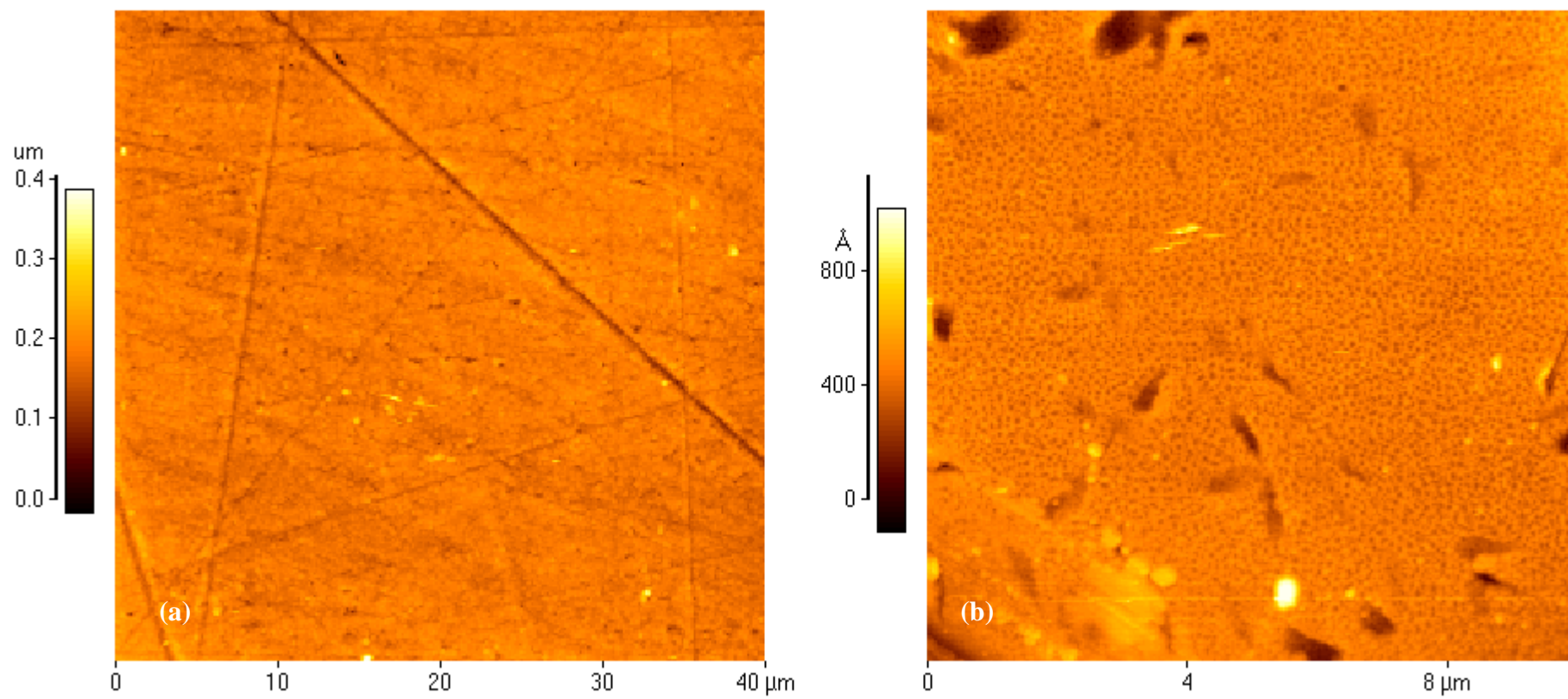


Fig. 16 AFM images of typical lapped and release Ni surfaces.
(a) Lapped surface of Ni Sandia Logo, (b) Release surface of Ni square.

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